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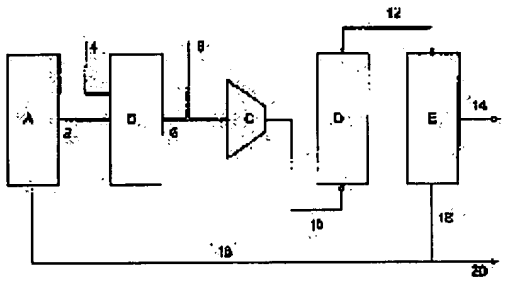
(54) METHOD FOR SEPARATING AND REMOVING PERFLUOROCARBONS FROM GAS STREAM

(57)Abstract:

PURPOSE: To provide an effective method for removing perfluorocarbons from a gas stream by adsorption.

CONSTITUTION: Prefluorocarbons are recovered from a gas stream by supplying the gas stream to an adsorption process using one or two or more energetically uniform adsorbent beds such as a high silicone adsorbent to FAU structure, a high silicone adsorbent of BEA structure and a high silicone adsorbent of MOR structure. As the adsorption process, a pressure swing adsorption or a temp. swing adsorption is preferable.



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CLAIMS

[Claim(s)]
[Claim 1] It is the approach of separating said at least one sort of gas perfluoro-carbonizing-ized hydrogen from the gas flow containing perfluoro-carbonizing-ized hydrogen, one sort, or two sorts or more of permanent gas. Said gas flow The high silicon adsorbent of FAU structure, the high silicon adsorbent of BEA structure, The high silicon adsorbent of MOR structure, the carbon molecular sieve which has the diameter of a hole of 4.5 Angstrom units at least, How to adsorb said at least one sort of perfluoro-carbonizing-ized hydrogen from said gas flow by through and it at the carbonized sulfonation styrene-divinylbenzene copolymer, the inside **** silicate of M41 S-structure class, and the adsorbent chosen from such mixture.
[Claim 2] Said at least one sort of gas perfluoro-carbonizing-ized hydrogen is a method according to claim 1 of having 1-8 carbon atoms.
[Claim 3] Said at least one sort of gas perfluoro-carbonizing-ized hydrogen is an approach according to claim 2 chosen from tetrafluoromethane, hexafluoroethane, an oct fluoro propane, tetrafluoroethylenes, and such mixture.
[Claim 4] Said one sort or two sorts or more of permanent gas is an approach according to claim 1 chosen from nitrogen, oxygen, argons, and such mixture.
[Claim 5] Said one sort or two sorts or more of permanent gas is an approach according to claim 3 chosen from nitrogen, oxygen, argons, and such mixture.
[Claim 6] Said adsorbent is the approach according to claim 1 of being the high silicon adsorbent of FAU structure.
[Claim 7] Said adsorbent is the approach according to claim 6 of being Y mold zeolite of a dealuminization mold.
[Claim 8] Said dealuminization type of Y mold zeolite is a method according to claim 7 of having the silicon pair aluminum ratio of about 100 at least.
[Claim 9] It is the circuit system adsorption approach for separating one sort or two sorts or more of gas perfluoro-carbon from the gas flow containing nitrogen. The :(a) aforementioned gas flow The high silicon adsorbent of FAU structure, the high silicon adsorbent of BEA structure, the high silicon adsorbent of MOR structure, The carbon molecular sieve which has the diameter of a hole of 4.5 Angstrom units at least, The carbonized sulfonation styrene-divinylbenzene copolymer, the inside **** silicate of M41 S-structure class, To at least one adsorbent floor chosen from such mixture, and through, How to consist of carrying out desorption of one sort or two sorts or more of gas perfluoro-carbon in which stuck to said one sort or two sorts or more of gas perfluoro-carbon from said gas flow, and (b) adsorption was subsequently carried out by it from said adsorbent.
[Claim 10] Said circuit system adsorption approach is an approach according to claim 9 chosen from a pressure-swing-adsorption method, temperature swing adsorption processes, and such combination.
[Claim 11] The adsorption process of said circuit system adsorption approach is [about]. -It is the approach according to claim 9 performed with the temperature of the range of 100 - 100 degrees C of abbreviation, and the absolute pressure of the range of about 0.5-20 bars.
[Claim 12] Said one sort or two sorts or more of gas perfluoro-carbon is the approach according to claim 9 chosen from tetrafluoromethane, hexafluoroethane, an oct fluoro propane, tetrafluoroethylenes, and such mixture.
[Claim 13] Said adsorbent is the approach according to claim 12 of being Y mold zeolite of a dealuminization mold.
[Claim 14] Said dealuminization type of Y mold zeolite is a method according to claim 13 of having the silicon pair aluminum ratio of about 100 at least.
[Claim 15] The gas mixture object which is the approach of defecating ** containing the chemical residue by silicon steam treatment, and contains a resultant, oxygen, and unreacted perfluoro-carbon for the perfluoro-carbon and oxygen of :(a) gas by through and it in ** is formed.;
(b) Introduce the inert gas chosen from nitrogen, argons, and such mixture into said gas mixture object.;
With said resultant, in the reactor containing the compound which reacts said gas mixture object (c) Through, It

removes said resultant from said gas mixture object substantially. As it does not have remarkable effect on said perfluoro-carbon in this case, however, the gas mixture object of; and (d) above which does not include the resultant substantially The high silicon adsorbent of FAU structure, the high silicon adsorbent of BEA structure, the high silicon adsorbent of MOR structure, The carbon molecular sieve which has the diameter of a hole of 4.5 Angstrom units at least, The carbonized sulfonation styrene-divinylbenzene copolymer, the inside **** silicate of M41 S-structure class, and; which presents the adsorption process of a circuit system using at least one adsorbent floor chosen from such mixture, and separates said perfluoro-carbon from the gas mixture object which does not include the resultant substantially [the above] by it -- an approach including each [these] process.

[Claim 16] The approach according to claim 15 of including further making the aforementioned room carry out recycling of the separated perfluoro-carbon.

[Claim 17] The approach according to claim 15 of including further carrying out scrubbing of said gas mixture object in advance of a process (c) using the solvent for at least one sort of resultants included in said gas mixture object.

[Claim 18] The aforementioned room is the approach according to claim 15 of being a vacuum evaporationo room or a silicon chip etching chamber.

[Claim 19] Said adsorbent is the approach according to claim 15 of being Y mold zeolite of a dealuminization mold.

[Claim 20] Said dealuminization type of Y mold zeolite is a method according to claim 19 of having the silicon pair aluminum ratio of about 100 at least.

[Claim 21] It is the approach of carrying out the polymerization of the tetrafluoroethylene, :(a) tetrafluoroethylene is contacted for a catalyst in a polymerization reactor, and the generation mixture which contains polytetrafluoroethylene and unreacted tetrafluoroethylene by it is formed.;

(b) Strip said generation mixture using inert gas, form the gas mixture object which contains stripping gas and unreacted tetrafluoroethylene by it and by which stripping was carried out, and the gas mixture object of; and (c) above by which stripping was carried out The high silicon adsorbent of FAU structure, the high silicon adsorbent of BEA structure, the high silicon adsorbent of MOR structure, The carbon molecular sieve which has the diameter of a hole of 4.5 Angstrom units at least, The carbonized sulfonation styrene-divinylbenzene copolymer, the inside **** silicate of M41 S-structure class, and; which presents the adsorption process of a circuit system using at least one adsorbent floor chosen from such mixture, and separates unreacted tetrafluoroethylene from the gas mixture object with which the stripping of the above was carried out by it -- an approach including each [these] process.

[Claim 22] The approach according to claim 21 of including further carrying out recycling of the unreacted tetrafluoroethylene from a process (c) to said polymerization reactor.

[Claim 23] Said adsorbent is the approach according to claim 21 of being Y mold zeolite of a dealuminization mold.

[Claim 24] Said dealuminization type of Y mold zeolite is a method according to claim 23 of having the silicon pair aluminum ratio of about 100 at least.

[Claim 25] Said inert gas is an approach according to claim 21 chosen from nitrogen, argons, and such mixture.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Industrial Application] Especially this invention relates to the approach adsorption removes perfluoro-carbonizing-ized hydrogen from gas flow, about purification of gas flow.
[0002]
[Background of the Invention] The hydrocarbon derivative (perfluoro-carbon) fully fluoro-ized (fluorination) is globally used in various home use and the industrial use field. Current emission of the gas perfluoro-carbonizing-ized hydrogen is carried out into the environment as a result of such use. As an example, the perfluoro-carbon of low molecular weight is used combining oxygen in the semi-conductor manufacture for etching of a silicon chip, and purification of a chemical-vacuum-deposition room. These processes are typically performed under a vacuum. The exhaust gas of ****** includes [others / perfluoro-carbon] an unreacted vacuum evaporationo compound and various resultants, such as hydrogen fluoride and 3 nitrogen fluoride. Since these compounds cannot be emitted to insurance into atmospheric air, exhaust gas is processed in order to change it to the compound which generally destroys the compound considered to be harmful, or can emit them into atmospheric air. According to one procedure, gas flow is introduced into a reactor (manufacturer: Edwards High Vacuum International Division of BOC Group (trade name: EDWARDS glass fiber reinforced cement)), for example, a gas reactor, and the component of gas flow reacts at an elevated temperature in it, and it is changed into destroyable solid matter. However, perfluoro-carbon is nonresponsiveness at altitude and it passes a reactor, without being influenced. Since it is not thought to the ozone layer which perfluoro-carbon is avirulent and surround the earth that it is harmful, they are emitted into current atmospheric air.
[0003] However, it is thought that perfluoro-carbon is what carries out warming of the earth on account of those advanced stability and a thermal property (global warmers). or [therefore, / that the industrial world in the world minimum-izes emission of the perfluoro-carbonizing-ized hydrogen to the inside of current and an environment] -- or the efforts for for stopping are performed. The process available now although these compounds are collected from abandonment gas is considered in how to destroy [costs increase, and] them, since it is not necessarily practical. In order to destroy, one proposed approach is combustion. This is performed by heating them in temperature of 1000 degrees C or more, and hydrogen and oxygen are given to the temperature by burning under existence of perfluoro-carbon.
[0004]
[Problem(s) to be Solved by the Invention] Destruction of perfluoro-carbon is not the best solution over the problem of abandonment. That is because costs increase and other harmful by-products arise by incomplete combustion. Furthermore, perfluoro-carbon is products with high value, and if it can collect them from gas flow at the costs which are not high, it will be beneficially reusable. In order to attain this purpose, this invention is effective in costs and offers an efficient approach.
[0005]
[Means for Solving the Problem] According to the large mode of this invention, one sort or two sorts or more of gas perfluoro-carbon is separated from said gas flow containing at least one sort of permanent gas by letting gas flow pass to the adsorbent (mesoporous) of inside **** of the microporosity adsorbent of one sort or two sorts or more of specific high silicon (high silicon) homogeneous in energy and/or one sort, or two sorts or more of specification homogeneous in energy. Perfluoro-carbon is adsorbed more strongly than the component of others of gas flow by these adsorbents. Perfluoro-carbon is recovered from an adsorbent by the usual playback procedure.
[0006] A desirable adsorbent is the mordenite (mordenite) of Y mold zeolite of a dealuminization mold, the beta mold zeolite of a dealuminization mold, and a dealuminization mold, and a silicon pair aluminum ratio is 50 or more about

these all. The silicon pair aluminum ratio of the most desirable adsorbent is Y mold zeolite of 100 or more dealuminization molds.
[0007] The approach of this invention can be used for collecting all the perfluoro-carbon of the condition of a gas or a steam in adsorption temperature. Especially this invention is especially suitable for recovery of saturation or ethylene partial saturation perfluoro-carbon, and suitable for recovery of the perfluoro-carbon, for example, perfluoro-methane, which has the carbon atom to eight pieces, perfluoro-ethane, perfluoro-ethylene, a perfluoro-hexane, a perfluoro-octane, etc.
[0008] Generally specifying the adsorption process used can use all adsorption procedures rather than it is important in this invention. Generally the adsorption process of a circuit system is used and the cycles of a pressure-swing-adsorption method (PSA) and a temperature swing adsorption process (TSA) or such combination are desirable. In two or three beds or more which became the lot arranged in parallel, when adsorption is presented with at least one floor, as for adsorption, it is desirable to be operated by the non-aligning formula so that another floor may be reproduced.
[0009] In addition to a mere adsorption process which was mentioned above, this invention can be used for changing a specific circulation treatment process. There are vacuum deposition, an etching chamber purification process, and a perfluoro-ethylene polymerization-ized process in the specific circulation treatment process which can incorporate this invention.
[0010] In vacuum evaporationo and purification of an etching chamber, ** containing a vacuum evaporationo object or an etching chemical-vacuum-deposition object is defecated by introducing perfluoro-carbon and oxygen under plasma conditions into **. Perfluoro-carbon and oxygen react with a vacuum evaporationo object, and form various gas products. a resultant, an unreacted vacuum evaporationo chemical, perfluoro-carbon, and the gas mixture object that consists of oxygen are taken out from a vacuum evaporationo room by exhaust air desirable, and is diluted with nitrogen or an argon by arbitration, and, subsequently to a reactor, it lets it pass at an elevated temperature. A vacuum evaporationo chemical changes with them to a harmless solid-state. Although perfluoro-carbon does not change within a reactor, it is picked out from a gas reactor distillate by the above-mentioned adsorption process, and recycling is carried out to a vacuum evaporationo room or an etching chamber, or it is stored for future use. Perfluoro-carbon can be given to further purification process like condensation or low temperature distillation, when the purity needs to be raised.
[0011] In the polymerization of perfluoro-ethylene, perfluoro-ethylene carries out a polymerization in a reactor, and a polymer and the mixed product of an unreacted monomer arise by it. An unreacted monomer is preferably removed from a mixed product by stripping using inert gas. Subsequently, the monomer by which stripping was carried out is given to an above-mentioned adsorption process, and is adsorbed by it in perfluoro-ethylene. After desorption is carried out from a bed, recycling of the perfluoro-ethylene is carried out to a polymerization reactor, or it is sent to a storage area or disposal is carried out.
[0012]
[The desirable mode for inventing] Perfluoro-carbon is separated from one or two permanent gas or more by the approach of this invention. Nitrogen, oxygen, an argon, helium, neon, a krypton, a xenon, hydrogen, and a carbon monoxide are contained in permanent gas on the purpose of this invention. Especially this invention is suitable for separating perfluoro-carbon from nitrogen, oxygen, argons, and such mixture.
[0013] It is the specific matter homogeneous in energy, and there is matter of the microporosity which has the diameter of a hole of about 4.5 Angstrom units at least, and inside **** in the adsorbent which can be used by this invention. On the purpose of this explanation, with the microporosity matter, it is defined as the matter which has the hole size of an average of less than about 20 Angstrom units, and is defined as the matter which has average hole size in the range of about 20 - about 500 Angstrom units with the matter of inside ****. In an adsorbent homogeneous in energy, the adsorption energy of all adsorption locations is substantially equivalent. this definition means coming out so, even if the heat of measurable adsorption is substantially fixed experimentally and it is a time of the concentration of the adsorbed compound changing. It is used here. "The heat of adsorption fixed on a real target" means that the heat of adsorption of the above-mentioned matter does not change at about 10% or more.
[0014] The thing of Y mold of the reforming FAU structure class zeolite of high silicon [adsorbent / suitable / microporosity], for example, a dealuminization mold,; the mordenite of the reforming BEA structure class zeolite of quantity silicon, for example, beta mold zeolite [of a dealuminization mold];, and the reforming MOR structure class zeolite of high silicon, for example, a dealuminization mold, and the thing which has the diameter of a hole of about 4.5 Angstrom units at least by the carbon molecular sieve (CMS), ******.
[0015] It is used here. The word "quantity silicon (silicon-rich)" means that the silicon pair aluminum ratio of a molecular sieve is about 50 or more. In the most desirable mode, the silicon pair aluminum ratio of a molecular sieve is about 100:1 or more. A suitable high silicon molecular sieve can be manufactured direct composition or by

dealuminization-izing the molecular sieve of a desired class. The method of preparing a high silicon molecular sieve and them is learned well, and those structures and manufacture approaches do not constitute any parts of this invention.
[0016] In the suitable adsorbent of inside ****, they are a polymerization coal property adsorbent, for example, sulfonation (it carbonized) styrene [which was pyrolyzed partially]-divinylbenzene copolymer (for example, Rohm andHaas product marketed with trademark of Ambersorb from shrine);, and the inside **** silicate of M41 S-structure class, and ******.
[0017] Perfluoro-carbon separable [with the process of this invention] is the things of the quality of a steam in the temperature and the pressure by which it is a gas thing, i.e., a gas thing or an adsorption process is usually carried out in ambient temperature and atmospheric pressure. The word "perfluoro-carbonizing-ized hydrogen" means the aliphatic hydrocarbon derivative by which all the hydrogen atoms were permuted by the fluorine atom. The saturation and ethylene partial saturation perfluoro-carbon which have the boiling point about 100 degrees C or less are one of those are contained in the compound of this class, and the perfluoro-carbon which has eight or less carbon atoms is contained in it. As a typical example of perfluoro-carbon recoverable [with the process of this invention], there are perfluoro-methane, perfluoro-ethane, a perfluoro-propane, a perfluoro-hexane, a perfluoro-octane, perfluoro-ethylene, etc.
[0018] With reference to an accompanying drawing, it explains so that this invention may be understood well. In the drawing, the same reference mark shows the same or similar component of equipment. The auxiliary device which is not required for an understanding of this invention, for example, a compressor, the heat exchanger, the bulb, etc. are excluded from the drawing, in order to give explanation of this invention plain.
[0019] If drawing 1 is referred to, in A, a vacuum means and D show a reactor and, as for a perfluoro-carbon storage container and B, E shows an adsorber, as for the vacuum deposition room of one or a lot or an etching chamber, and C. The configuration of these units and the detail of actuation are known well, and they do not constitute any parts of this invention.
[0020] Perfluoro-carbon and oxygen are supplied to ** B through Rhine 2 and 4, respectively, and ** is exhausted through Rhine 6. Rhine 6 connects ** B with the entry of the vacuum means C. The vacuum means C is a vacuum pump typically. The vacuum means C is connected with Reactor D through Rhine 10 by part for the trailer. Reactor D has the component of a gas process stream, one sort which reacts, or two sorts or more of matter, and a means (not shown) for heating this gas reactor to desired reaction temperature. The details of the gas reactor D do not constitute any parts of this invention, therefore they are not clarified in this explanation. The gas reactor D is connected with Unit E by Rhine 12 in the outlet edge.
[0021] Abandonment gas discharge Rhine 14 and perfluoro-carbon discharge Rhine 16 are established in Unit E. Rhine 16 is connected with perfluoro-carbon recycling Rhine 18 (this is prepared in order to return the purified perfluoro-carbon to Container A), and perfluoro-carbon discharge Rhine 20 in the illustrated example.
[0022] Various gassing units, for example, filter, or solvent-cleaning scrubbers are arranged between Units D and E or to Rhine 14 between the units C and D of equipment, and you may make it remove the component of particulate matter or fusibility from equipment by request. However, since it is not important about this invention, they are not illustrated.
[0023] The main purposes of Unit E are separating perfluoro-carbon from the gas distillate from the gas reactor D. Typically, Unit E is pressure-swing-adsorption equipment or a temperature swing adsorber, and consists of two or more put floors which was preferably filled up with one sort or two sorts or more of molecular-sieve adsorbents of a class homogeneous in energy mentioned above. Generally a floor is arranged in parallel, and it is adjusted so that it may operate by the cyclic process which consists of adsorption and desorption. Usually, while the equipment with which adsorption is performed consists of two or more beds of a non-aligning formula and it is operating in the adsorption phase where one or two beds or more are cycles so that it may be carried out, one or other two beds or more are reproduced.
[0024] In case the approach of this invention shown in drawing 1 is enforced, perfluoro-carbon and oxygen let Rhine 2 and 4 pass, respectively, and it is introduced into ** B of the equipment which chemical vacuum deposition or etching actuation just completed. **** -- various chemical abandonment vacuum evaporationo matter is included, and it is necessary to remove those matter from ** so that ** may be prepared for the following chemical vacuum deposition or etching actuation Perfluoro-carbon and oxygen contact the abandonment vacuum evaporationo matter, and react with them, and a gas abandonment product generates them. A gas product is taken out from a vacuum evaporationo room by suction produced with the vacuum means C with unreacted perfluoro-carbon and oxygen. Since a gas product and unreacted oxygen form the mixture of a flammability, in order to prevent early combustion of generation gas, inert gas, for example, nitrogen, an argon, and a carbon dioxide are introduced into Rhine 6 through Rhine 8. Subsequently to the inside of Reactor D the gas mixture object which passed through Rhine 10 is introduced, and mixture is heated by about 600 degrees C or more in it. The various components of the heated mixture contact reacting matter in a column, and

change to the product which can discharge safely into an environment or can be easily collected by future chemical preparation. The unreacted perfluoro-carbon in Rhine 10 passes Unit D without changing. Subsequently to Equipment E the effluent gas from a gas reactor enters, and receives gas adsorption in it.

[0025] An adsorption process consists of repeating an adsorption process and the playback process of a floor. In a desirable mode, an adsorption process is pressure swing adsorption, temperature swing adsorption, or these two combination, and the specific adsorption approach is determined by the chemical composition of process gas. Although the specific conditions at the time of an adsorption process being carried out determine the effectiveness of an adsorption process, they do not constitute a part of this invention. These conditions are well known to this contractor of the field of a gas adsorption process, and all the combination of the actuation conditions which are used in an adsorption process and which change widely can be used by the approach of this invention.

[0026] Generally, an adsorption process is [about] usually. -It is carried out from less than [100 degrees C or it] with the temperature of the range to about +100 degrees C, and the absolute pressure of the range to about 0.5 to about 20 bars, and is preferably carried out with the temperature of the range from about 15 degrees C to about 75 degrees C, and the absolute pressure of the range to about 1 to about 10 bars. The separability ability of an adsorbent is so good that temperature is low. Distributed gas is introduced into an adsorber between the adsorption processes of a process, and it flows each inside of the floor in the adsorption phase of a cycle. When gas flows the inside of a floor, an adsorbent is adsorbed in perfluoro-carbon. When an adsorption process advances, the front of the adsorption formed in the front edge of the adsorbed perfluoro-carbon moves forward in the direction of the outlet of the gas by which it does not adsorb. The remainder of gas flow passes through a floor and comes out of Equipment E through Rhine 14 as abandonment gas. Abandonment gas may be discharged in atmospheric air, supposing it does not contain the component harmful to an environment. Or it may be sent to a downstream device for the further processing. When the front of adsorption arrives at the location of a request of a bed, the flow of the distributed gas to the inside of a floor stops. This shows termination of the adsorption phase of a separation process.

[0027] The floor which completed the adsorption phase receives playback next. Similarly conditions to perform playback of a floor are not important, in order to obtain a good result in operation of this invention. Although playback of a PSA floor can be performed with the absolute pressure of about 100mb or less, it is usually performed with the absolute pressure of the range of about 100 to about 1000mb. playback of a TSA floor is performed by heating an adsorbent to temperature higher than the temperature to which an adsorption process is carried out -- having -- the temperature -- typical -- the temperature of the range of about 0 to about 200 degrees C -- it is the temperature of the range of about 20 to about 150 degrees C preferably. desorption -- a heater -- and/or, it can carry out by letting a steam or the heated inert gas pass in a floor. The pressure between the playback processes of a TSA cycle and in an adsorption container is comparable as the pressure currently maintained within the container between adsorption processes, or can be made lower than it. It is often desirable to perform a temperature swing process in atmospheric pressure or its near. When the combination of a pressure-swing-adsorption cycle and a temperature swing adsorption cycle is used, temperature is high and a pressure is lower than the time of a cycle being in an adsorption process between the playback processes of a floor.

[0028] Desorption of the perfluoro-carbon is carried out through Rhine 16 during playback from Unit E. If the collected perfluoro-carbon is the thing of purity suitable for it carrying out recycling to equipment, it can be returned to a storage container A through Rhine 18. Or it can be discharged from equipment through Rhine 20 for the further purification.

[0029] In the mode of this invention shown in drawing 2 , in F, a polymerization reactor and G show a polymer recovery unit, and H shows a tetrafluoroethylene adsorber. All these units and equipment are known well and the concrete details of those configurations and actuation do not constitute a part of this invention.

[0030] The monomer supply line 30 and polymer discharge Rhine 32 are attached to Reactor F. These neither is illustrated, although Reactor F is a tetrafluoroethylene polymerization reactor of a typical batch type or continuous system, and it is equipped with the means for heating the means for agitating the contents of a reactor, and the contents of a reactor etc. while all the standard descriptions required for polymerization-izing of a tetrafluoroethylene monomer, for example, a catalyst supply line, and a polymerization are performed.

[0031] Polymer discharge Rhine 32 is connected to the entry of the polymer recovery unit G. Unit G is a stripping unit typically and stripping gas entry Rhine 34, polymer recovery Rhine 36, and outlet Rhine 38 of gas by which stripping was carried out are attached to it. or [that the adsorption unit H is similar to the unit E of drawing 1 although Rhine 38 is connected to the entry of the adsorption unit H] -- or it is the same. The abandonment gas line 40 and tetrafluoroethylene recovery Rhine 42 are attached to Unit H.

[0032] In case this invention is carried out with the equipment of drawing 2 , the additive of a request of tetrafluoroethylene and others, for example, a catalyst, a polymer modifier, etc., is introduced into Reactor F through

Rhine 30 or another supply line. Polymerization-ization can be performed to a batch type or continuous system in the liquid phase or a gaseous phase. The mixture of a polymer product and an unreacted monomer is picked out from Reactor F through Rhine 32, and is introduced into the polymer recovery unit G. In Unit G, the stripping of the polymer is carried out using inert gas, for example, nitrogen, or an argon, and the stripping of the unreacted monomer is carried out by it from a polymer. The polymer by which stripping was carried out is taken out from Unit G, and it is sent to the further processing unit on the lower stream of a river of the equipment of drawing 2 , and the gas flow containing stripping gas and unreacted tetrafluoroethylene by which stripping was carried out is discharged from Unit G through Rhine 38, and, subsequently to the inside of the adsorption recovery unit H, is introduced. In Unit H, unreacted tetrafluoroethylene is the approach mentioned above about the equipment of drawing 1 , and is adsorbed from supply flow. Subsequently, desorption of the adsorbed tetrafluoroethylene is carried out from an adsorbent, when a polymerization process is the thing of continuous system, recycling of it is carried out through Rhine 42 to Reactor F, or when a polymerization process is the thing of a batch type, it is sent to a tetrafluoroethylene storage container.

[0033]

[Example] Although the following examples explain this invention further, unless it is shown especially, the section, the percentage, and the ratio are expressed with volume criteria.

[0034] example one diameter of 1/4 inch (6.35mm) -- a gas chromatograph with a die length of 2 feet (61cm) -- the column was filled up with the pellet (this is marketed with the trademark of a Degussa Wessalith DAY mold zeolite from Degussa AG) with which extrusion molding of the Y mold zeolite of a dealuminization mold was carried out. It let the helium as carrier gas pass in the column by the flow rate of 30 ml/min using the column maintained by the temperature of 0 degree C. The sample of the gas mixture object which consists of nitrogen, 1% of tetrafluoromethane (CF4), and 2% of hexafluoroethane (C2F6) was poured in into carrier gas, and it let it pass in the column filled up with the adsorbent. The elution time amount of each component is shown in Table 1. The separation factor of CF4/N2 and C2F6/N2 about each trial is measured, and these also show in Table 1. The above-mentioned procedure was repeated in the temperature of 20 degrees C and 30 degrees C. The result of these trials is also shown in Table 1.

[0035]

[Table 1]

試験No.	吸着温度 (℃)	滞留時間（分）			分離係数	
		N_2	CF_4	C_2F_6	CF_4/N_2	C_2F_6/N_2
1	0	0.67	2.96	—	4.42	—
2	20	0.57	2.10	15.29	3.68	26.83
3	30	0.52	1.58	11.06	3.07	21.48

[0036] Example In 2 this example, a series of pressure-swing-adsorption trials were performed using various nitrogen-hexafluoroethane (C2F6) gas mixture objects as a feed stock. the supply flow about trials 1, 2, 3, and 5 -- nitrogen and C2F6 from -- it becomes. The supply flow about trial 4 is nitrogen and C2F6. 4% of oxygen is included in others. All trials were performed by die length of 20 inches (50.8cm) in the adsorption container of the cylindrical shape of a pair with a diameter of 1.25 inches (31.8mm). The bed was filled up with the Degussa Wessalith DAY mold zeolite in trials 1-4, and the floor was filled up with Ambersorb 563 (trademark) adsorbent in the trial 5. The adsorption container of each other was operated at a non-aligning ceremony by the adsorption cycle of 30 minutes. Equalization of the pressurization =3 second; adsorption =447 second; floor this cycle of whose is a feed stock, equalization of a reduced pressure =3 second; floor of an exhaust air =447 second; floor, backfilling (backfill) = 3 seconds of a recompression =3 second; product; it has each process. In equalization of a floor, in the trial of 1, 2, and 5, equalization of a floor went for hard flow to the forward direction in (top-to-top) and the trial of 3 and 4 (top-to-bottom). It was carried out by various temperature and flow rates which adsorption is performed by the pressure of 3.77 bars, and are shown in Table 2. The adsorption container was exhausted to the absolute pressure of 100mb with the vacuum pump between exhaust air processes. Adsorption temperature, a supply flow rate (a part for liter/), the concentration of the hexafluoroethane in distributed gas flow and product gas flow, and C2F6 Recovery is shown in Table 2 (trials 1-4).

[0037]

[Table 2]

試験 No.	吸着温度 (℃)	供給流量 (l/m)	供給流れ中の C_2F_6 の濃度 (%)	生成物流れ中の C_2F_6 の濃度 (%)	C_2F_6 の回収率 (%)
1	25	0.3	2.21	11.74	93.99
2	5	0.3	3.05	13.35	98.95
3	5	0.5	9.76	49.00	87.29
4	25	0.25	5.09	22.93	93.39
5	25	0.50	1.31	5.3	98.44

[0038] Although this invention was explained with the specific experiment in a specific example, it does not pass over an example to mere instantiation of this invention, but it is thought that it is possible. [of various modification] For example, a bed can be constituted from mixture of two sorts or three sorts or more of adsorbents of an above-mentioned type, or two sorts or three sorts or more of adsorbents can also be put in order and used for a serial. Furthermore, this invention can only be carried out as an adsorption process or a part of process of others desirable although perfluoro-carbon is collected again as a part of defecation process of a vacuum evaporationo room, or etching process. For example, it can use in order to collect the perfluoro-carbon formed between aluminum purification actuation in this invention, in order to separate perfluoro-carbon from the gas for laser (lasing gas), or in order to collect coolant gas. The range of this invention is limited by only the claim.

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TECHNICAL FIELD

[Industrial Application] Especially this invention relates to the approach adsorption removes perfluoro-carbonizing-ized hydrogen from gas flow, about purification of gas flow.
[0002]
[Background of the Invention] The hydrocarbon derivative (perfluoro-carbon) fully fluoro-ized (fluorination) is globally used in various home use and the industrial use field. Current emission of the gas perfluoro-carbonizing-ized hydrogen is carried out into the environment as a result of such use. As an example, the perfluoro-carbon of low molecular weight is used combining oxygen in the semi-conductor manufacture for etching of a silicon chip, and purification of a chemical-vacuum-deposition room. These processes are typically performed under a vacuum. The exhaust gas of ****** includes [others / perfluoro-carbon] an unreacted vacuum evaporationo compound and various resultants, such as hydrogen fluoride and 3 nitrogen fluoride. Since these compounds cannot be emitted to insurance into atmospheric air, exhaust gas is processed in order to change it to the compound which generally destroys the compound considered to be harmful, or can emit them into atmospheric air. According to one procedure, gas flow is introduced into a reactor (manufacturer: Edwards High Vacuum International Division of BOC Group (trade name: EDWARDS glass fiber reinforced cement)), for example, a gas reactor, and the component of gas flow reacts at an elevated temperature in it, and it is changed into destroyable solid matter. However, perfluoro-carbon is nonresponsiveness at altitude and it passes a reactor, without being influenced. Since it is not thought to the ozone layer which perfluoro-carbon is avirulent and surround the earth that it is harmful, they are emitted into current atmospheric air.
[0003] However, it is thought that perfluoro-carbon is what carries out warming of the earth on account of those advanced stability and a thermal property (global warmers). or [therefore, / that the industrial world in the world minimum-izes emission of the perfluoro-carbonizing-ized hydrogen to the inside of current and an environment] -- or the efforts for for stopping are performed. The process available now although these compounds are collected from abandonment gas is considered in how to destroy [costs increase, and] them, since it is not necessarily practical. In order to destroy, one proposed approach is combustion. This is performed by heating them in temperature of 1000 degrees C or more, and hydrogen and oxygen are given to the temperature by burning under existence of perfluoro-carbon.

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TECHNICAL PROBLEM

[Problem(s) to be Solved by the Invention] Destruction of perfluoro-carbon is not the best solution over the problem of abandonment. That is because costs increase and other harmful by-products arise by incomplete combustion. Furthermore, perfluoro-carbon is products with high value, and if it can collect them from gas flow at the costs which are not high, it will be beneficially reusable. In order to attain this purpose, this invention is effective in costs and offers an efficient approach.

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MEANS

[Means for Solving the Problem] According to the large mode of this invention, one sort or two sorts or more of gas perfluoro-carbon is separated from said gas flow containing at least one sort of permanent gas by letting gas flow pass to the adsorbent (mesoporous) of inside **** of the microporosity adsorbent of one sort or two sorts or more of specific high silicon (high silicon) homogeneous in energy and/or one sort, or two sorts or more of specification homogeneous in energy. Perfluoro-carbon is adsorbed more strongly than the component of others of gas flow by these adsorbents. Perfluoro-carbon is recovered from an adsorbent by the usual playback procedure.
[0006] A desirable adsorbent is the mordenite (mordenite) of Y mold zeolite of a dealuminization mold, the beta mold zeolite of a dealuminization mold, and a dealuminization mold, and a silicon pair aluminum ratio is 50 or more about these all. The silicon pair aluminum ratio of the most desirable adsorbent is Y mold zeolite of 100 or more dealuminization molds.
[0007] The approach of this invention can be used for collecting all the perfluoro-carbon of the condition of a gas or a steam in adsorption temperature. Especially this invention is especially suitable for recovery of saturation or ethylene partial saturation perfluoro-carbon, and suitable for recovery of the perfluoro-carbon, for example, perfluoro-methane, which has the carbon atom to eight pieces, perfluoro-ethane, perfluoro-ethylene, a perfluoro-hexane, a perfluoro-octane, etc.
[0008] Generally specifying the adsorption process used can use all adsorption procedures rather than it is important in this invention. Generally the adsorption process of a circuit system is used and the cycles of a pressure-swing-adsorption method (PSA) and a temperature swing adsorption process (TSA) or such combination are desirable. In two or three beds or more which became the lot arranged in parallel, when adsorption is presented with at least one floor, as for adsorption, it is desirable to be operated by the non-aligning formula so that another floor may be reproduced.
[0009] In addition to a mere adsorption process which was mentioned above, this invention can be used for changing a specific circulation treatment process. There are vacuum deposition, an etching chamber purification process, and a perfluoro-ethylene polymerization-ized process in the specific circulation treatment process which can incorporate this invention.
[0010] In vacuum evaporationo and purification of an etching chamber, ** containing a vacuum evaporationo object or an etching chemical-vacuum-deposition object is defecated by introducing perfluoro-carbon and oxygen under plasma conditions into **. Perfluoro-carbon and oxygen react with a vacuum evaporationo object, and form various gas products. a resultant, an unreacted vacuum evaporationo chemical, perfluoro-carbon, and the gas mixture object that consists of oxygen are taken out from a vacuum evaporationo room by exhaust air desirable, and is diluted with nitrogen or an argon by arbitration, and, subsequently to a reactor, it lets it pass at an elevated temperature. A vacuum evaporationo chemical changes with them to a harmless solid-state. Although perfluoro-carbon does not change within a reactor, it is picked out from a gas reactor distillate by the above-mentioned adsorption process, and recycling is carried out to a vacuum evaporationo room or an etching chamber, or it is stored for future use. Perfluoro-carbon can be given to further purification process like condensation or low temperature distillation, when the purity needs to be raised.
[0011] In the polymerization of perfluoro-ethylene, perfluoro-ethylene carries out a polymerization in a reactor, and a polymer and the mixed product of an unreacted monomer arise by it. An unreacted monomer is preferably removed from a mixed product by stripping using inert gas. Subsequently, the monomer by which stripping was carried out is given to an above-mentioned adsorption process, and is adsorbed by it in perfluoro-ethylene. After desorption is carried out from a bed, recycling of the perfluoro-ethylene is carried out to a polymerization reactor, or it is sent to a storage area or disposal is carried out.
[0012]
[The desirable mode for inventing] Perfluoro-carbon is separated from one or two permanent gas or more by the

approach of this invention. Nitrogen, oxygen, an argon, helium, neon, a krypton, a xenon, hydrogen, and a carbon monoxide are contained in permanent gas on the purpose of this invention. Especially this invention is suitable for separating perfluoro-carbon from nitrogen, oxygen, argons, and such mixture.

[0013] It is the specific matter homogeneous in energy, and there is matter of the microporosity which has the diameter of a hole of about 4.5 Angstrom units at least, and inside **** in the adsorbent which can be used by this invention. On the purpose of this explanation, with the microporosity matter, it is defined as the matter which has the hole size of an average of less than about 20 Angstrom units, and is defined as the matter which has average hole size in the range of about 20 - about 500 Angstrom units with the matter of inside ****. In an adsorbent homogeneous in energy, the adsorption energy of all adsorption locations is substantially equivalent. this definition means coming out so, even if the heat of measurable adsorption is substantially fixed experimentally and it is a time of the concentration of the adsorbed compound changing. It is used here. "The heat of adsorption fixed on a real target" means that the heat of adsorption of the above-mentioned matter does not change at about 10% or more.

[0014] The thing of Y mold of the reforming FAU structure class zeolite of high silicon [adsorbent / suitable / microporosity], for example, a dealuminization mold,; the mordenite of the reforming BEA structure class zeolite of quantity silicon, for example, beta mold zeolite [of a dealuminization mold];, and the reforming MOR structure class zeolite of high silicon, for example, a dealuminization mold, and the thing which has the diameter of a hole of about 4.5 Angstrom units at least by the carbon molecular sieve (CMS), ******.

[0015] It is used here. The word "quantity silicon (silicon-rich)" means that the silicon pair aluminum ratio of a molecular sieve is about 50 or more. In the most desirable mode, the silicon pair aluminum ratio of a molecular sieve is about 100:1 or more. A suitable high silicon molecular sieve can be manufactured direct composition or by dealuminization-izing the molecular sieve of a desired class. The method of preparing a high silicon molecular sieve and them is learned well, and those structures and manufacture approaches do not constitute any parts of this invention.

[0016] In the suitable adsorbent of inside ****, they are a polymerization coal property adsorbent, for example, sulfonation (it carbonized) styrene [which was pyrolyzed partially]-divinylbenzene copolymer (for example, Rohm andHaas product marketed with trademark of Ambersorb from shrine);, and the inside **** silicate of M41 S-structure class, and ******.

[0017] Perfluoro-carbon separable [with the process of this invention] is the things of the quality of a steam in the temperature and the pressure by which it is a gas thing, i.e., a gas thing or an adsorption process is usually carried out in ambient temperature and atmospheric pressure. The word "perfluoro-carbonizing-ized hydrogen" means the aliphatic hydrocarbon derivative by which all the hydrogen atoms were permuted by the fluorine atom. The saturation and ethylene partial saturation perfluoro-carbon which have the boiling point about 100 degrees C or less are one of those are contained in the compound of this class, and the perfluoro-carbon which has eight or less carbon atoms is contained in it. As a typical example of perfluoro-carbon recoverable [with the process of this invention], there are perfluoro-methane, perfluoro-ethane, a perfluoro-propane, a perfluoro-hexane, a perfluoro-octane, perfluoro-ethylene, etc.

[0018] With reference to an accompanying drawing, it explains so that this invention may be understood well. In the drawing, the same reference mark shows the same or similar component of equipment. The auxiliary device which is not required for an understanding of this invention, for example, a compressor, the heat exchanger, the bulb, etc. are excluded from the drawing, in order to give explanation of this invention plain.

[0019] If drawing 1 is referred to, in A, a vacuum means and D show a reactor and, as for a perfluoro-carbon storage container and B, E shows an adsorber, as for the vacuum deposition room of one or a lot or an etching chamber, and C. The configuration of these units and the detail of actuation are known well, and they do not constitute any parts of this invention.

[0020] Perfluoro-carbon and oxygen are supplied to ** B through Rhine 2 and 4, respectively, and ** is exhausted through Rhine 6. Rhine 6 connects ** B with the entry of the vacuum means C. The vacuum means C is a vacuum pump typically. The vacuum means C is connected with Reactor D through Rhine 10 by part for the trailer. Reactor D has the component of a gas process stream, one sort which reacts, or two sorts or more of matter, and a means (not shown) for heating this gas reactor to desired reaction temperature. The details of the gas reactor D do not constitute any parts of this invention, therefore they are not clarified in this explanation. The gas reactor D is connected with Unit E by Rhine 12 in the outlet edge.

[0021] Abandonment gas discharge Rhine 14 and perfluoro-carbon discharge Rhine 16 are established in Unit E. Rhine 16 is connected with perfluoro-carbon recycling Rhine 18 (this is prepared in order to return the purified perfluoro-carbon to Container A), and perfluoro-carbon discharge Rhine 20 in the illustrated example.

[0022] Various gassing units, for example, filter, or solvent-cleaning scrubbers are arranged between Units D and E or to Rhine 14 between the units C and D of equipment, and you may make it remove the component of particulate matter

or fusibility from equipment by request. However, since it is not important about this invention, they are not illustrated.
[0023] The main purposes of Unit E are separating perfluoro-carbon from the gas distillate from the gas reactor D. Typically, Unit E is pressure-swing-adsorption equipment or a temperature swing adsorber, and consists of two or more put floors which was preferably filled up with one sort or two sorts or more of molecular-sieve adsorbents of a class homogeneous in energy mentioned above. Generally a floor is arranged in parallel, and it is adjusted so that it may operate by the cyclic process which consists of adsorption and desorption. Usually, while the equipment with which adsorption is performed consists of two or more beds of a non-aligning formula and it is operating in the adsorption phase where one or two beds or more are cycles so that it may be carried out, one or other two beds or more are reproduced.
[0024] In case the approach of this invention shown in drawing 1 is enforced, perfluoro-carbon and oxygen let Rhine 2 and 4 pass, respectively, and it is introduced into ** B of the equipment which chemical vacuum deposition or etching actuation just completed. **** -- various chemical abandonment vacuum evaporationo matter is included, and it is necessary to remove those matter from ** so that ** may be prepared for the following chemical vacuum deposition or etching actuation Perfluoro-carbon and oxygen contact the abandonment vacuum evaporationo matter, and react with them, and a gas abandonment product generates them. A gas product is taken out from a vacuum evaporationo room by suction produced with the vacuum means C with unreacted perfluoro-carbon and oxygen. Since a gas product and unreacted oxygen form the mixture of a flammability, in order to prevent early combustion of generation gas, inert gas, for example, nitrogen, an argon, and a carbon dioxide are introduced into Rhine 6 through Rhine 8. Subsequently to the inside of Reactor D the gas mixture object which passed through Rhine 10 is introduced, and mixture is heated by about 600 degrees C or more in it. The various components of the heated mixture contact reacting matter in a column, and change to the product which can discharge safely into an environment or can be easily collected by future chemical preparation. The unreacted perfluoro-carbon in Rhine 10 passes Unit D without changing. Subsequently to Equipment E the effluent gas from a gas reactor enters, and receives gas adsorption in it.
[0025] An adsorption process consists of repeating an adsorption process and the playback process of a floor. In a desirable mode, an adsorption process is pressure swing adsorption, temperature swing adsorption, or these two combination, and the specific adsorption approach is determined by the chemical composition of process gas. Although the specific conditions at the time of an adsorption process being carried out determine the effectiveness of an adsorption process, they do not constitute a part of this invention. These conditions are well known to this contractor of the field of a gas adsorption process, and all the combination of the actuation conditions which are used in an adsorption process and which change widely can be used by the approach of this invention.
[0026] Generally, an adsorption process is [about] usually. -It is carried out from less than [100 degrees C or it] with the temperature of the range to about +100 degrees C, and the absolute pressure of the range to about 0.5 to about 20 bars, and is preferably carried out with the temperature of the range from about 15 degrees C to about 75 degrees C, and the absolute pressure of the range to about 1 to about 10 bars. The separability ability of an adsorbent is so good that temperature is low. Distributed gas is introduced into an adsorber between the adsorption processes of a process, and it flows each inside of the floor in the adsorption phase of a cycle. When gas flows the inside of a floor, an adsorbent is adsorbed in perfluoro-carbon. When an adsorption process advances, the front of the adsorption formed in the front edge of the adsorbed perfluoro-carbon moves forward in the direction of the outlet of the gas by which it does not adsorb. The remainder of gas flow passes through a floor and comes out of Equipment E through Rhine 14 as abandonment gas. Abandonment gas may be discharged in atmospheric air, supposing it does not contain the component harmful to an environment. Or it may be sent to a downstream device for the further processing. When the front of adsorption arrives at the location of a request of a bed, the flow of the distributed gas to the inside of a floor stops. This shows termination of the adsorption phase of a separation process.
[0027] The floor which completed the adsorption phase receives playback next. Similarly conditions to perform playback of a floor are not important, in order to obtain a good result in operation of this invention. Although playback of a PSA floor can be performed with the absolute pressure of about 100mb or less, it is usually performed with the absolute pressure of the range of about 100 to about 1000mb. playback of a TSA floor is performed by heating an adsorbent to temperature higher than the temperature to which an adsorption process is carried out -- having -- the temperature -- typical -- the temperature of the range of about 0 to about 200 degrees C -- it is the temperature of the range of about 20 to about 150 degrees C preferably. desorption -- a heater -- and/or, it can carry out by letting a steam or the heated inert gas pass in a floor. The pressure between the playback processes of a TSA cycle and in an adsorption container is comparable as the pressure currently maintained within the container between adsorption processes, or can be made lower than it. It is often desirable to perform a temperature swing process in atmospheric pressure or its near. When the combination of a pressure-swing-adsorption cycle and a temperature swing adsorption cycle is used,

temperature is high and a pressure is lower than the time of a cycle being in an adsorption process between the playback processes of a floor.

[0028] Desorption of the perfluoro-carbon is carried out through Rhine 16 during playback from Unit E. If the collected perfluoro-carbon is the thing of purity suitable for it carrying out recycling to equipment, it can be returned to a storage container A through Rhine 18. Or it can be discharged from equipment through Rhine 20 for the further purification.

[0029] In the mode of this invention shown in drawing 2 , in F, a polymerization reactor and G show a polymer recovery unit, and H shows a tetrafluoroethylene adsorber. All these units and equipment are known well and the concrete details of those configurations and actuation do not constitute a part of this invention.

[0030] The monomer supply line 30 and polymer discharge Rhine 32 are attached to Reactor F. These neither is illustrated, although Reactor F is a tetrafluoroethylene polymerization reactor of a typical batch type or continuous system, and it is equipped with the means for heating the means for agitating the contents of a reactor, and the contents of a reactor etc. while all the standard descriptions required for polymerization-izing of a tetrafluoroethylene monomer, for example, a catalyst supply line, and a polymerization are performed.

[0031] Polymer discharge Rhine 32 is connected to the entry of the polymer recovery unit G. Unit G is a stripping unit typically and stripping gas entry Rhine 34, polymer recovery Rhine 36, and outlet Rhine 38 of gas by which stripping was carried out are attached to it. or [that the adsorption unit H is similar to the unit E of drawing 1 although Rhine 38 is connected to the entry of the adsorption unit H] -- or it is the same. The abandonment gas line 40 and tetrafluoroethylene recovery Rhine 42 are attached to Unit H.

[0032] In case this invention is carried out with the equipment of drawing 2 , the additive of a request of tetrafluoroethylene and others, for example, a catalyst, a polymer modifier, etc., is introduced into Reactor F through Rhine 30 or another supply line. Polymerization-ization can be performed to a batch type or continuous system in the liquid phase or a gaseous phase. The mixture of a polymer product and an unreacted monomer is picked out from Reactor F through Rhine 32, and is introduced into the polymer recovery unit G. In Unit G, the stripping of the polymer is carried out using inert gas, for example, nitrogen, or an argon, and the stripping of the unreacted monomer is carried out by it from a polymer. The polymer by which stripping was carried out is taken out from Unit G, and it is sent to the further processing unit on the lower stream of a river of the equipment of drawing 2 , and the gas flow containing stripping gas and unreacted tetrafluoroethylene by which stripping was carried out is discharged from Unit G through Rhine 38, and, subsequently to the inside of the adsorption recovery unit H, is introduced. In Unit H, unreacted tetrafluoroethylene is the approach mentioned above about the equipment of drawing 1 , and is adsorbed from supply flow. Subsequently, desorption of the adsorbed tetrafluoroethylene is carried out from an adsorbent, when a polymerization process is the thing of continuous system, recycling of it is carried out through Rhine 42 to Reactor F, or when a polymerization process is the thing of a batch type, it is sent to a tetrafluoroethylene storage container.

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EXAMPLE

[Example] Although the following examples explain this invention further, unless it is shown especially, the section, the percentage, and the ratio are expressed with volume criteria.
[0034] example one diameter of 1/4 inch (6.35mm) -- a gas chromatograph with a die length of 2 feet (61cm) -- the column was filled up with the pellet (this is marketed with the trademark of a Degussa Wessalith DAY mold zeolite from Degussa AG) with which extrusion molding of the Y mold zeolite of a dealuminization mold was carried out. It let the helium as carrier gas pass in the column by the flow rate of 30 ml/min using the column maintained by the temperature of 0 degree C. The sample of the gas mixture object which consists of nitrogen, 1% of tetrafluoromethane (CF4), and 2% of hexafluoroethane (C2F6) was poured in into carrier gas, and it let it pass in the column filled up with the adsorbent. The elution time amount of each component is shown in Table 1. The separation factor of CF4/N2 and C2F6/N2 about each trial is measured, and these also show in Table 1. The above-mentioned procedure was repeated in the temperature of 20 degrees C and 30 degrees C. The result of these trials is also shown in Table 1.
[0035]
[Table 1]

試験No.	吸着温度 (℃)	滞留時間（分）			分離係数	
		N_2	CF_4	C_2F_6	CF_4/N_2	C_2F_6/N_2
1	0	0.67	2.96	—	4.42	—
2	20	0.57	2.10	15.29	3.68	26.83
3	30	0.52	1.58	11.06	3.07	21.48

[0036] Example In 2 this example, a series of pressure-swing-adsorption trials were performed using various nitrogen-hexafluoroethane (C2F6) gas mixture objects as a feed stock. the supply flow about trials 1, 2, 3, and 5 -- nitrogen and C2F6 from -- it becomes. The supply flow about trial 4 is nitrogen and C2F6. 4% of oxygen is included in others. All trials were performed by die length of 20 inches (50.8cm) in the adsorption container of the cylindrical shape of a pair with a diameter of 1.25 inches (31.8mm). The bed was filled up with the Degussa Wessalith DAY mold zeolite in trials 1-4, and the floor was filled up with Ambersorb 563 (trademark) adsorbent in the trial 5. The adsorption container of each other was operated at a non-aligning ceremony by the adsorption cycle of 30 minutes. Equalization of the pressurization =3 second; adsorption =447 second; floor this cycle of whose is a feed stock, equalization of a reduced pressure =3 second; floor of an exhaust air =447 second; floor, backfilling (backfill) = 3 seconds of a recompression =3 second; product; it has each process. In equalization of a floor, in the trial of 1, 2, and 5, equalization of a floor went for hard flow to the forward direction in (top-to-top) and the trial of 3 and 4 (top-to-bottom). It was carried out by various temperature and flow rates which adsorption is performed by the pressure of 3.77 bars, and are shown in Table 2. The adsorption container was exhausted to the absolute pressure of 100mb with the vacuum pump between exhaust air processes. Adsorption temperature, a supply flow rate (a part for liter/), the concentration of the hexafluoroethane in distributed gas flow and product gas flow, and C2F6 Recovery is shown in Table 2 (trials 1-4).
[0037]
[Table 2]

試験 No.	吸着温度 (℃)	供給流量 (l/m)	供給流れ中の C_2F_6 の濃度 (%)	生成物流れ中の C_2F_6 の濃度 (%)	C_2F_6 の回収率 (%)
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DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]
[Drawing 1] It is the block diagram showing one example of the equipment for enforcing the approach of this invention.

[Drawing 2] It is the block diagram showing another example of the equipment for enforcing the approach of this invention.

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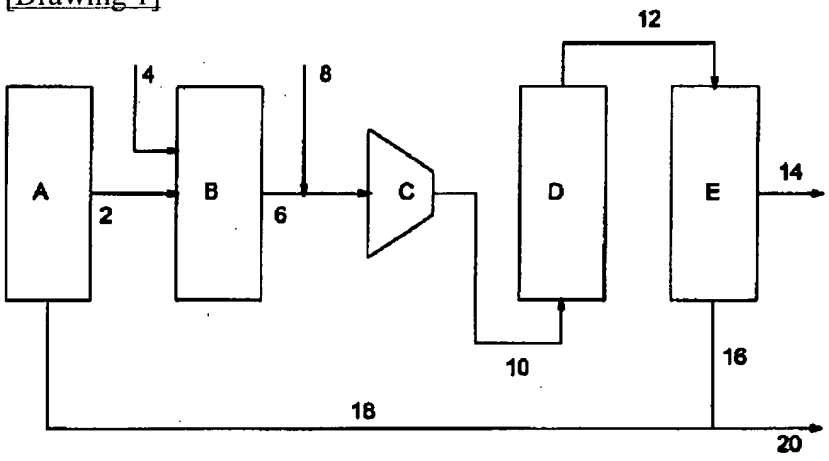
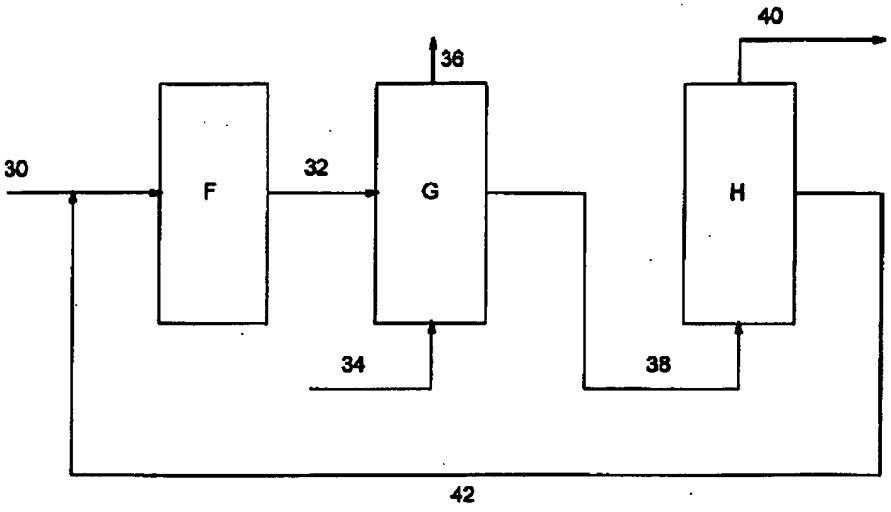
DRAWINGS

[Drawing 1]



[Drawing 2]



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